

This Page Is Inserted by IFW Operations
and is not a part of the Official Record

BEST AVAILABLE IMAGES

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images may include (but are not limited to):

- BLACK BORDERS
- TEXT CUT OFF AT TOP, BOTTOM OR SIDES
- FADED TEXT
- ILLEGIBLE TEXT
- SKEWED/SLANTED IMAGES
- COLORED PHOTOS
- BLACK OR VERY BLACK AND WHITE DARK PHOTOS
- GRAY SCALE DOCUMENTS

IMAGES ARE BEST AVAILABLE COPY.

**As rescanning documents *will not* correct images,
please do not report the images to the
Image Problem Mailbox.**

THIS PAGE BLANK (USPTO)

09381890



The
Patent
Office

PCT/GB 98 / 0 0 9 5 0

#13

REC'D 3 0 APR 1998

The Patent Office PCT
Cardiff Road
Newport
Gwent
NP9 1RH

I, the undersigned, being an officer duly authorised in accordance with Section 74(1) and (4) of the Deregulation and Contracting Out Act 1994, to sign and issue certificates on behalf of the Comptroller-General, hereby certify that annexed hereto is a true copy of the documents as originally filed in connection with the patent application identified therein.

In accordance with the Patents (Companies Re-registration) Rules 1982, if a company named in this certificate and any accompanying documents has re-registered under the Companies Act 1980 with the same name as that with which it was registered immediately before re-registration save for the substitution as, or inclusion as, the last part of the name of the words "public limited company" or their equivalents in Welsh, references to the name of the company in this certificate and any accompanying documents shall be treated as references to the name with which it is so re-registered.

In accordance with the rules, the words "public limited company" may be replaced by p.l.c., plc, P.L.C. or PLC.

Re-registration under the Companies Act does not constitute a new legal entity but merely subjects the company to certain additional company law rules.

PRIORITY DOCUMENT

Signed

Andrew Gersy

Dated

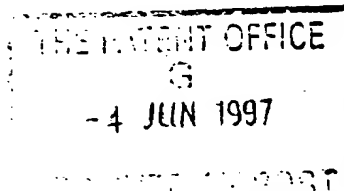
24 MAR 1998

THIS PAGE BLANK (USFTO)

04JUN97 E279116-1 002903
P01/7700 25.00 - 9711447.4

Request for grant of a patent

(See the notes on the back of this form. You can also get an explanatory leaflet from the Patent Office to help you fill in this form)



The Patent Office

Cardiff Road
Newport
Gwent NP9 1RH

1. Your reference	10155P2GB/EAI	4 JUN 1997
2. Patent application number (The Patent Office will fill in this part)	9711447.4	
3. Full name, address and postcode of the or of each applicant (underline all surnames)	Reckitt & Colman France 15 Rue Ampère BP 83 - 91301 MASSY CEDEX France Patents ADP number (if you know it) If the applicant is a corporate body, give the country/state of its incorporation	
	4912424001 France	
4. Title of the invention	Improvements in or relating to Depilatory Materials	
5. Name of your agent (if you have one)	E. A. Ilott	
"Address for service" in the United Kingdom to which all correspondence should be sent (including the postcode)	Reckitt & Colman plc Group Patents Department Dansom Lane HULL HU8 7DS 649220153	
	Patents ADP number (if you know it)	
6. If you are declaring priority from one or more earlier patent applications, give the country and the date of filing of the or of each of these earlier applications and (if you know it) the or each application number	Country European (filed in France)	Priority application number (if you know it) 97400811.2
		Date of filing (day / month / year) 9 April 1997
7. If this application is divided or otherwise derived from an earlier UK application, give the number and the filing date of the earlier application	Number of earlier application	Date of filing (day / month / year)
8. Is a statement of inventorship and of right to grant of a patent required in support of this request? (Answer 'Yes' if: a) any applicant named in part 3 is not an inventor, or b) there is an inventor who is not named as an applicant, or c) any named applicant is a corporate body. See note (d))	Yes	

Patents Form 1/77

9. Enter the number of sheets for any of the following items you are filing with this form. Do not count copies of the same document

Continuation sheets of this form	...
Description	10
Claim(s)	03
Abstract	01
Drawing(s)	03

10. If you are also filing any of the following, state how many against each item.

Priority documents	...
Translations of priority documents	N/A
Statement of inventorship and right to grant of a patent (Patents Form 7/77)	...
Request for preliminary examination and search (Patents Form 9/77)	1
Request for substantive examination (Patents Form 10/77)	...
Any other documents (please specify)	Fee Sheet (FS1)

11.

I/We request the grant of a patent on the basis of this application.

Signature

E. A. Ilott

Date 2 June 1997

Agent for the Applicant

12. Name and daytime telephone number of person to contact in the United Kingdom

Liz Ilott

01482 582909

Warning

After an application for a patent has been filed, the Comptroller of the Patent Office will consider whether publication or communication of the invention should be prohibited or restricted under Section 22 of the Patents Act 1977. You will be informed if it is necessary to prohibit or restrict your invention in this way. Furthermore, if you live in the United Kingdom, Section 23 of the Patents Act 1977 stops you from applying for a patent abroad without first getting written permission from the Patent Office unless an application has been filed at least 6 weeks beforehand in the United Kingdom for a patent for the same invention and either no direction prohibiting publication or communication has been given, or any such direction has been revoked.

Notes

- If you need help to fill in this form or you have any questions, please contact the Patent Office on 0645 500505.
- Write your answers in capital letters using black ink or you may type them.
- If there is not enough space for all the relevant details on any part of this form, please continue on a separate sheet of paper and write "see continuation sheet" in the relevant part(s). Any continuation sheet should be attached to this form.
- If you have answered 'Yes' Patents Form 7/77 will need to be filed.
- Once you have filled in the form you must remember to sign and date it.
- For details of the fee and ways to pay please contact the Patent Office.

IMPROVEMENTS IN OR RELATING TO DEPILATORY MATERIALS

This invention relates to depilatory materials.

5 Materials for removing superfluous body hair are well known. These are of various types. One type of material requires to be initially heated before being applied to the skin in a generally molten state. It is then allowed to solidify before being removed from the
10 skin together with unwanted hair. Another type of material is in the form of a cream containing a substance which degrades hair keratin.

 Depilatory materials in the form of gels are less common and attempts to market such gels have had only
15 limited success. The gels tended to have a higher skin irritancy than cream depilatories and became very sticky when being rinsed off because of the nature of the polymer present in the formulation. Moreover, the materials needed to have a high pH value and problems
20 were encountered in formulating a gel which would be stable in such highly alkaline conditions.

 It is an object of the present invention to provide a depilatory material in the form of a gel which does not suffer from these disadvantages.

25 It is a further object of the present invention to provide a depilatory material in the form of a gel which may be sprayed onto the skin without propellant.

 It is another object of the present invention to provide a depilatory gel having a reduced tendency to
30 irritate the skin.

 It has been found that these objects can be achieved by forming the gel from two water soluble polymeric components in association and in a pH environment maintained by a preferred buffer system.

35 One of the polymeric components provides the gel with appropriate viscosity and the other polymeric component

contributes to the stability of the gel.

Accordingly, one aspect of the present invention provides a depilatory material in the form of an aqueous alkaline gel comprising:-

- 5 (i) a polymeric binder comprising a water soluble first component which is a charged cross-linked water soluble polymer and a water soluble second component which is a linear non-ionic polymer and/or a positively charged polymer and/or a negatively charged polymer,
- 10 (ii) a substance capable of degrading hair keratin, and
- (iii) a buffer system capable of maintaining the pH of the material at a value of from 10.5 from
- 15 13.0.

The cross-linked polymer of the first component is such that it swells at high pH and may be, for example, a cross-linked acrylic polymer such as a carbomer, for example, Ultrez 10 and/or Carbopol ETD 2020 from

20 Goodrich and Structure 2000 and 3001 from National Starch. Another suitable polymer is a cross-linked polyacrylamide such as Seppigel 305. Preferably, the polymer is a copolymer of methyl vinyl ether and maleic anhydride cross-linked with dodecadiene available from

25 ISP under the trade designation Antaron Stabilise 06. Preferably, the depilatory material includes from 0.1 to 3.0% by weight of the first component.

The second component may be present in the material in an amount of from 0.2 to 3.0% by weight and

30 it may comprise a water soluble nonionic polymer such as polyethylene oxide or polyethylene glycol (PEG), an hydrophobically modified polyol such as Acrysol 44 from Rohm and Haas, or a poly vinyl pyrrolidone such as PVP K from ISP or Luviskol from BASF. Alternatively, or

35 additionally, the second component may comprise a natural water soluble polymer such as cellulose,

hydroxy propyl cellulose, cetyl hydroxyethyl or propyl cellulose (for example Natrosol Plus from Aqualon), starch, polyglucoside, a polysaccharide such as an alginate, guar gum, carrageenan (lambda, kappa, iota),
5 a sclerotium gum, a polypeptide with or without any alkyl chain (C_1 to C_{18}) modification and cationic end groups such as quaternary ammonium groups with counter ions such as chlorides and bromides, or a copolymer of sodium acrylate and dimethyl diallyl ammonium chloride
10 (such as that known as Merquat), or a copolymer of hydroxy ethyl cellulose and dimethyl diallyl ammonium chloride (such as that known as Celquat).

Preferably, the second component comprises polyethylenimine (such as that known under the trade
15 name Lupasol from BASF) and/or polyvinyl pyrrolidone (such as that known under the trade designation PVP K-30 from ISP).

These exemplified polymers, when combined in accordance with the present invention, form a binder
20 having unique viscosity and elasticity, sustained high ionic strength and high pH.

The substance capable of degrading keratin may be, for example, a sulphur compound such as potassium thioglycolate or thioglycerol. Also, the material may
25 include components which will accelerate the keratin reduction reaction such as urea, thiourea or dithioerythritol, dimethyl isosorbide (DMI), ethoxydiglycol (transcutol) and methyl propyldiol (MP diol). DMI is preferred.

30 It is important that the pH of the material is from about 10.5 to 13, and preferably about 12.0 to 12.5, in order to achieve good depilation within about 5 minutes. In order to achieve this, a suitable buffer should be present such as sodium silicate, L-arginine,
35 sodium nicotinate, potassium phosphate or polyethylenimine preferably in combination with urea

and thioglycolate.

Generally, the buffer system will be present in an amount of from about 2.0 to 6.0% by weight. Advantageously the buffer system comprises from 2.0 to 3.0% by wt (based on the total material) of L-arginine and from 0 to 1.0% by wt (based on the total material) of polyethylenimine.

The pH buffer system is important to ensure efficacy of the depilation within 5 minutes whilst also minimizing skin irritation. In order to achieve minimum skin irritation a buffer system comprising L-arginine and/or polyethylenimine, urea and potassium and/or dipotassium thioglycolate is preferred.

The gels are transparent depending on the amount of active ingredients present and on the procedure of mixing. They have a thick rich feel and excellent rinsability with water. Moreover it is not necessary to include a surfactant and hence they are less likely to cause skin damage.

Due to the special rheology of the polymeric binder, it is possible to include beads, dyes and/or oily particles which will remain dispersed in the gel. The oily particles may, for example, include azulene or jojoba polyethylene glycol ester to reduce irritation, preferably in combination with L-arginine, urea and potassium thioglycolate or thioglycerol. The beads may be encapsulated algae extract and sweet almond oil.

Although it is possible to provide a gel in accordance with the present invention which has viscoelastic properties suitable for spraying, the depilatory material may alternatively be applied by means of a roll-on device or as a shower gel packaging.

According to another aspect of the present invention there is provided a process for producing a depilatory material as defined above which comprises:-

- (i) forming a first premix by mixing water and

the first component a second premix by mixing water, the second component and the buffer, and a third premix by mixing said substance and a keratin degradation accelerator,

5 (ii) adding the third premix to the second premix with stirring,

 (iii) adding the first premix to the resultant mixture with stirring, and

 (iv) adding alkali to obtain a pH of 10.5 to 13.0.

10 For a better understanding of the invention and to show how the same may be carried into effect, reference will now be made, by way of example, to the accompanying drawings, in which:-

 Figure 1 is a graph of shear stress (Pa) against shear rate (s^{-1}) for a series of polymeric binders for the materials of the invention,

 Figure 2 is a graph of the yield points (Pa) of the binders of Figure 1 against the cross-linked polymer contents (% w/w) of the binders, and

20 Figure 3 is a graph of the viscosities (Pas) of the binders of Figure 1 against the shear rate (s^{-1}) of the binders.

 The following Examples illustrate the invention:

25 EXAMPLES 1 TO 6

 A series of depilatory gels was prepared having the compositions shown in the following Table 1:-

TABLE 1

	1	2	3	4	5	6
Phase 1						
5 Polymer A	3.0%	2.5%	2.25%	2.0%	1.5%	1.0%
deionised water	47.0%	39.0%	36.25%	31.4%	23.5%	15.7%
Phase 2						
10 Polymer B	2.0%	2.0%	2.0%	2.0%	2.0%	2.0%
pH buffer	3.0%	3.0%	3.0%	3.0%	3.0%	3.0%
deionised water	10.7%	19.2%	24.8%	26.4%	38.3%	45.8%
15 Phase 3						
potassium thioglycolate (30%)	14.7%	14.7%	14.7%	14.7%	14.7%	14.7%
urea	8.0%	8.0%	8.0%	8.0%	8.0%	8.0%
20						
Final mix						
Phase 1	50.0%	41.5%	37.5%	33.4%	25.0%	16.7%
Phase 2	15.7%	24.2%	29.8%	33.4%	43.3%	50.8%
Phase 3	22.7%	22.7%	22.7%	22.7%	22.7%	22.7%
25 Potassium hydroxide 50%	11.6%	11.6%	10.0%	10.5%	9.0%	9.8%
Total	100.0%	100.0%	100.0%	100.0%	100.0%	100.0%
30 Total deionised water	57.5%	58.0%	59.9%	59.6%	61.6%	61.3%

In the table all amounts are expressed as % weight/weight; Polymer A was a methyl vinyl ether/maleic anhydride copolymer cross-linked with
5 dodecadiene known under the trade designation Antaron STO6 from ISP; and polymer B was polyvinyl pyrrolidone known under the trade designation PVP K-30 from ISP. The pH buffer system was a combination of 2% by wt of L-arginine and 1% by weight of polyethylenimine (based
10 on the total material weight).

In each case, an Olsa mixer with planar stirring and a high shear stirrer equipped with a vacuum and heating/cooling system was used. The procedure was as follows:-

15 Phase 1: Premix polymer A and water.

The water was placed in the mixer and polymer A was added slowly with stirring. When the polymer had been dispersed, heating was commenced whilst maintaining a vacuum of about 0.5 to 0.8 bar to avoid
20 air bubbles. The vacuum pump was operated intermittently in order to reduce water loss. When the temperature had reached about 80°C an almost clear gel had been formed. This was allowed to cool and swell overnight.

25 Phase 2: Premix polymer B, water, pH buffer

The water and polymer B were introduced into the mixer and homogenised for 2 minutes. The pH buffer system was then added and the mixture homogenised for a further 2 minutes.

30 Phase 3: Urea and potassium thioglycolate

The urea was added to the potassium thioglycolate in a beaker and stirred for 1 hour to complete dissolution.

The final mixing was effected as follows:-

35 Phase 2 was placed in the vessel and phase 3 was added with stirring for 15 minutes. A white

precipitate appeared. Phase 1 was added while mixing slowly under vacuum (0.5 bar) with planar stirring and the high shear stirrer operated at low speed. The mixture was homogenised for 30 minutes until a lumpy gel had been formed. The potassium hydroxide was then added. The gel became clear and transparent. The pH was adjusted, as necessary, to from 11.0 to 12.5.

Each of the resultant gels was subjected to rheological measurement. The parameters considered were shear stress, shear rate, viscosity and yield point. The results are shown in the Figures.

As can be seen from Figure 1, the variation of shear stress with shear rate is non-linear between 0 and 500 s^{-1} and thus the gel has a non-Newtonian behaviour with a yield point. The yield point is the stress required to make the gel flow. Figure 2 shows that the variation of the yield point (τ_0), i.e the shear stress for zero shear rate, varies exponentially. For a small variation in the amount of polymer A, the variation in the yield point, i.e. the variation in spreadability or consistency, may be significant.

The following Table 2 gives characteristic shear rates of products to be sprayed with a conventional pump spray and products to be applied from a shower gel packaging.

TABLE 2

Type of packaging	rate of product in cm^3/s	radius of the external output (cm)	Shear rate S^{-1}
Pump spray	0.17	0.0175 (radius of the actuator)	500-40000
shower gel packaging	8.8	ca 0.15	500-30000

The relationship between shear rate and viscosity

for the above mentioned samples is shown in Figure 3 from which it can be seen that, at the shear rates given in Table 2, the viscosity is very low.

5 It has been found that in order for the gel to be sprayable with a pump spray, its yield point should be not more than from 100 to 200 Pa. If, however, the gel is to be used in a shower gel pack, the yield point may be as high as 300Pa. Also, in order for the product to remain still for 5 minutes on the skin when held
10 vertical, the yield point needs to be about 60 Pa or more. Thus gels wherein polymer A constitutes from 2 to 2.5% w/w are preferred for spraying whereas rather lower percentages of polymer A may be present where the gel is to be used in the form of a shower gel
15 packaging.

In a further series of experiments, the blue powder Azulene was dissolved in thick paraffin oil to produce a 1% w/w solution. 2% w/w of this solution was then slowly added to each of the above gels and stirred
20 for 5 minutes. The Azulene remained stably suspended within the gel in the form of blue oily droplets. Depending upon the concentration of Azulene in the premix and the concentration of premix in the gel, the size and colour of the droplets and the overall
25 transparency of the gel could be adjusted.

In a further series of experiments, 0.5% w/w of Elespher was slowly added to each gel and stirred for 5 minutes. Elespher consists of encapsulated beads of algae extract and sweet almond oil and is green in
30 colour. The beads were stably suspended in the gel.

Example 7

Gels were made in accordance with the following Table 3:

TABLE 3

	% W/W
phase 1	
polymer A	1-3
water	40.00
KOH 50%	4.00
total phase 1	47.00
phase 2	
polymer B	0.6-3
buffer system	3.00
water	8.20
total phase 2	11.80
phase 3	
water	6.50
urea	8.00
potassium thioglycolate (30%)	14.70
total phase 3	29.20
water	1.85
KOH 50%	6.15

The amount of water was varied depending upon the amounts of polymers A and B present. Polymers A and B were as used in Examples 1 to 6 and the mixing technique employed was as described in those Examples. In each case, sprayable stable depilatory gels were obtained.

CLAIMS:

1. A depilatory material in the form of an aqueous alkaline gel comprising:
 - (i) a polymeric binder comprising a water soluble first component which is a charged cross-linked polymer and a water soluble second component which is a linear non-ionic polymer and/or a positively charged polymer and/or a negatively charged polymer,
 - (ii) a substance capable of degrading hair keratin, and
 - (iii) a system capable of maintaining the pH of the material at a value of from 10.5 to 13.0.
2. A depilatory material as claimed in claim 1, wherein the cross-linked polymer of the first component is a cross-linked acrylic polymer.
3. A depilatory material as claimed in claim 1, wherein the cross-linked copolymer of the first component is a copolymer of methyl vinyl ether and maleic anhydride cross-linked with dodecadiene.
4. A depilatory material as claimed in claim 1, 2, or 3 wherein the content of the first component is from 0.1 to 3.0 % w/w.
5. A depilatory material as claimed in any preceding claim, wherein the second component comprises a linear non-ionic polymer.
6. A depilatory material as claimed in claim 5, wherein the linear non-ionic polymer is polyvinyl pyrrolidone.
7. A depilatory material as claimed in claim 5, wherein the linear non-ionic polymer is polyethylene oxide or polyethylene glycol.
8. A depilatory material as claimed in claim 5, wherein the second component comprises cellulose, hydroxy propyl cellulose, cetyl hydroxy ethyl cellulose, cetyl propyl cellulose, starch, a polyglucoside, or a polysaccharide.

9. A depilatory material as claimed in any preceding claim, wherein the second component comprises sclerotium gum or a polypeptide.

5 10. A depilatory material as claimed in any preceding claim, wherein the second component comprises a polymer containing quaternary ammonium groups.

10 11. A depilatory material as claimed in claim 10, wherein the polymer of the second component is a copolymer of sodium acrylate or of hydroxy ethyl cellulose with dimethyl diallyl ammonium chloride.

12. A depilatory material as claimed in any preceding claim wherein the second component comprises polyethyleneimine.

15 13. A depilatory material as claimed in any preceding claim, wherein the content of the second component is from 0.2 to 3.0% by wt.

20 14. A depilatory material as claimed in any preceding claim, wherein the substance capable of degrading keratin is potassium thioglycolate or thioglycerol.

15. A depilatory material as claimed in any preceding claim and including an accelerator for the degradation of keratin.

25 16. A depilatory material as claimed in claim 15, wherein the accelerator is urea, thiourea, dithioerythritol, ethoxydiglycol or methylpropyldiol.

17. A depilatory material as claimed in claim 15, wherein the accelerator is dimethyl isosorbide.

30 18. A depilatory material as claimed in any preceding claim including a buffer system comprising a silicate, arginine, a nicotinate or a phosphate.

19. A depilatory material as claimed in any one of claims 1 to 17, wherein the buffer system comprises polyethylenimine.

35 20. A depilatory material as claimed in claim 19, wherein the buffer system additionally comprises L-

arginine.

21. A depilatory system as claimed in claim 19 or 20, wherein the buffer system additionally includes urea.

5 22. A depilatory system as claimed in claim 19, 20 or 21 wherein the buffer system additionally includes a thioglycolate.

23. A depilatory material as claimed in any preceding claim and having a yield point of not more
10 than 200 Pa.

24. A depilatory material as claimed in any one of claims 1 to 22 and having a yield point of not more than 300 Pa.

25. A depilatory material as claimed in any
15 preceding claim and having a yield point of at least 60 Pa.

26. A depilatory material as claimed in claim 1 substantially as hereinbefore described in any one of the foregoing Examples.

20 27. A process for producing a depilatory material as claimed in claim 1 which comprises:-

(i) forming a first premix by mixing water and the first component, a second premix by mixing water, the second component and the buffer system, and a third
25 premix by mixing said substance and a keratin degradation accelerator,

(ii) adding the third premix to the second premix with stirring,

(iii) adding the first premix to the resultant
30 mixture with stirring, and

(iv) adding alkali to obtain a pH of 10.5 to 13.0.

28. A process according to claim 27 substantially as hereinbefore described in the foregoing Examples.

29. A depilatory material whenever produced by
35 the process of claim 27 or 28.

ABSTRACT

IMPROVEMENTS IN OR RELATING TO DEPILATORY MATERIALS

5 A depilatory material comprises a substance
capable of degrading hair keratin in an aqueous gel
formed from a water soluble polymeric binder and
buffered to a pH of from 10.5 to 13.0. The polymeric
binder is formed from a first component which is a
charged cross-linked polymer and a second component
10 which comprises a non-ionic and/or charged polymer.
The gel is stable and can be applied by means of a pump
spray.

1/3

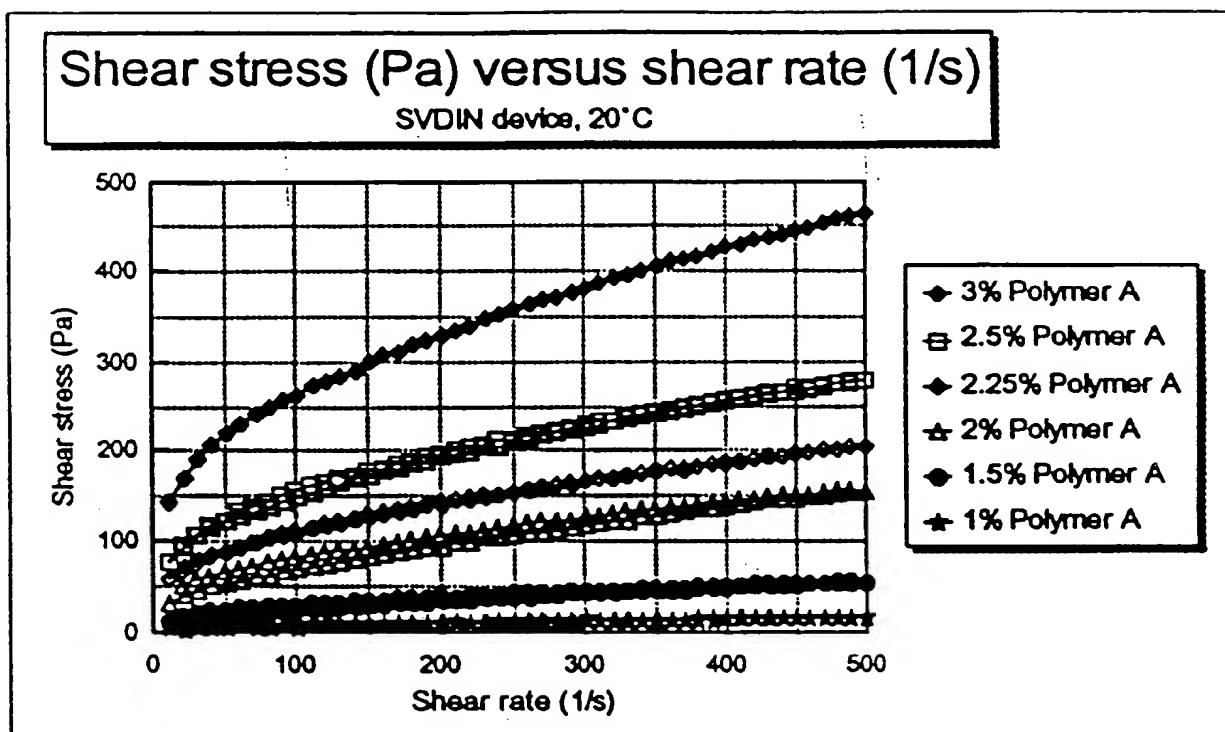


FIG. 1

THIS PAGE BLANK (USPTO)

2/3

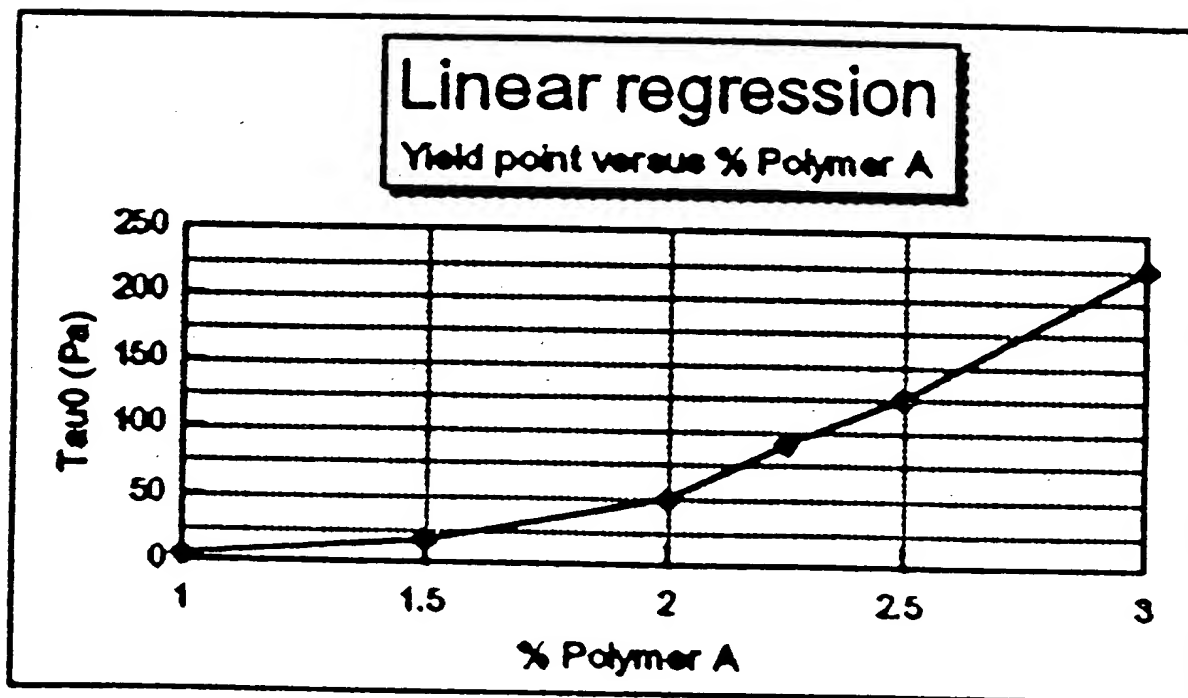


FIG. 2.

THIS PAGE BLANK (USPTO)

3/3

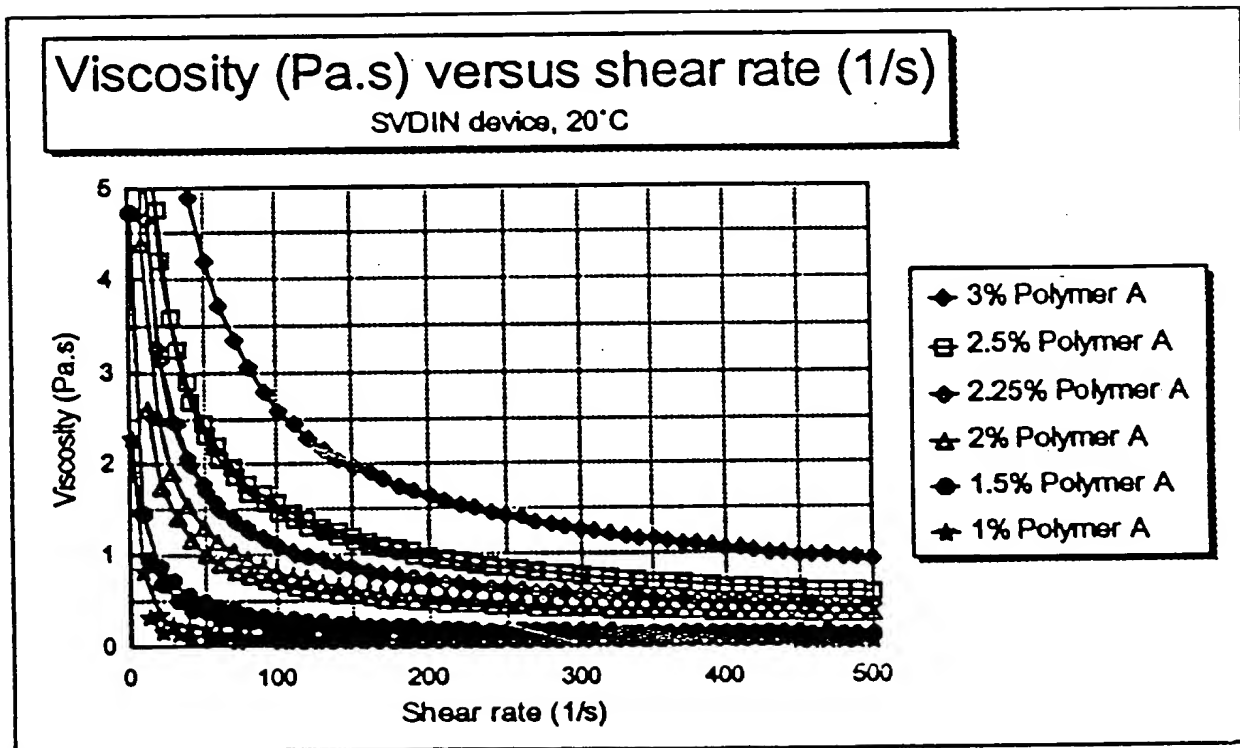


FIG. 3

THIS PAGE BLANK (USPTO)